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Date: 3 October 2001

BY COURIER

Mr Annick Canelas 53, rue de Voves 28630 Le Coudray FRANCE

Dear Mr Canelas

United States Patent Application No. 08291-619001 Sarah Desmots et al "Depilatory Compositions, Their Preparation and Use"

In order to proceed with the above United States patent for Project System H it is necessary to file the enclosed Combined Declaration and Power of Attorney at the United States Patent Office. I would be grateful if you would please sign and date this document where indicated and return the executed document to my attention in the addressed envelope provided. As we are not able to obtain stamps for the return envelope, we will be happy to reimburse you for the cost of return postage.

Please note that you are under obligation to sign this declaration by virtue of your former contractual rights with Reckitt & Colman France. You have already assigned your rights in this application to Reckitt & Colman France. The enclosed declaration is a formal requirement under the United States patent laws and we would greatly appreciate your urgent attention to this matter.

We would be grateful if you would also please inform us of any changes to your address. If your address has changed from that noted on the attached Declaration, please update the address details on the form in manuscript.

Please note that this document should be signed in <u>blue ink</u>. The reason for this is that, when documents are signed in black ink, it is sometimes queried whether the documents are originals or photocopies.

I would be grateful if you could complete this document and return to my attention prior to 15 October 2001. If you require any further information regarding the execution of this document, please do not hesitate to contact me directly on +44 1482 583730 or by e-mail to Amanda. Wilde@Reckittbenckiser.com





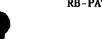
I thank you in anticipation of your prompt attention to this matter and look forward to receiving the executed document as a matter of extreme urgency.

Yours faithfully RECKITT BENCKISER plc

Amanda J. Wilde

Formalities & Records Manager

h:hq\patents\shared\ajw\crspndnc\10456-2





Attorney's Docket No.: 08291-619001 Client's Ref. No.: 10456P2-Usw

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I Annick CANELAS hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor, together with Bruno GUILLAUME, Philippe LEDON, David ACHER, Séverine POETTE, Gerd DAHMS, Sarah DESMOTS and Hubert DELAGNEAU, of the subject matter which is claimed and for which a patent is sought on the invention entitled DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE, the specification of which:

was filed on <u>September 24, 1999</u> as Application Serial No. <u>09/381,890</u> and, was described and claimed in PCT International Application No. <u>PCT/GB98/00950</u> filed on <u>30 March 1998</u>.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information I know to be material to patentability in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

| Country | Application No. | Filing Date | Priority Claimed |
|----------|-----------------|--------------|------------------|
| European | 97400811.2 | 9 April 1997 | [X] Yes [] No |
| British | 9711447.4 | 4 June 1997 | [X] Yes [] No |

I hereby appoint the following attorneys and/or agents to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

Frederick H. Rabin, Reg. No. 24,488; John B. Pegram, Reg. No. 25, 198; Richard P. Ferrara, Reg. No. 30,632 Samuel Borodach, Reg. No. 38,388 Andrew N. Parfomak, Reg. No. 32,431 William J. Hone, Reg. No. 26,739 Stephan J. Filipek, Reg. No. 33,384 Chris T. Mizumoto, Reg. No. 42,899

Address all telephone calls to FREDERICK H. RABIN at telephone number (212) 765-5070.

Address all correspondence to FREDERICK H. RABIN at:

FISH & RICHARDSON P.C. 45 Rockefeller Plaza, Suite 2800 New York, New York10111

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or

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2008/046



Attorney's Docket No.: 08291-619001 Client's Ref. No.: 10456P2-Usw

Combined Declaration and Power of Attorney Page 2 of 2 Pages

imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

| Full Name of Inventor: | ANNICK CANELAS | | |
|------------------------|--------------------|-------|---|
| Inventor's Signature: | | Date: | |
| Residence Address: | Le Coudray, France | | ÷ |
| Citizenship: | France | | |
| Post Office Address: | 53, rue de Voves | | |
| | 28630 Le Coudray | | |
| | FRANCE | | |

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:

Sarah DESMOTS et al.

Int'l Appln. No.

PCT/GB98/00950

Int'l Filing Date:

30 March 1998

U.S. Serial No.:

09/381,890

Title:

DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE

DECLARATION RE INVENTOR ANNICK CANELAS

I, Frederick H. Rabin, declare as follows:

- 1. I am an attorney of record in this application and am responsible for its prosecution.
- 2. In a renewed Petition under 37 C.F.R. § 1.47(a) filed on 21 February 2002, we provided evidence to show that inventor Annick Canelas had refused to sign a Declaration under 37 C.F.R. § 1.63. In a Decision on Petition dated 27 August 2002, this evidence was deemed insufficient because there was no clear showing that the declaration sent to Ms. Canelas was accompanied by a copy of the subject application.
- 3. In 20 November 2002, I sent a letter by airmail to Ms. Annick Canelas at her last known address asking her to execute a Combined Declaration and Power of Attorney. The Combined Declaration and Power of Attorney was stapled to a copy of PCT publication WO 98/44898. Airmail, rather than a faster courier service, was chosen in the expectation that, if Ms. Canelas was no longer at that address, the letter would be forwarded. Attached, as Exhibit N-1, is a copy of my letter to Ms. Canelas and a copy of the documents included with said letter.
- 4. I received no response from Ms. Canelas and my letter was not returned by the postal authorities.
- 5. On 18 December 2002, I sought the assistance of Ms. Karen R. Grant, Formalities Officer in the Group Patents Department of Reckitt Benckiser plc, the parent company of the current owner of the application, Reckitt Benckiser France.

Applicant: Sarah DESMO al al Serial No.: 09/381,890

Filed: September 24, 1999

Page: 2

- 6. In early January 2003, I was informed through a series of e-mails from Ms. Grant and Ms. Cristelle Leboucher-Madec of Reckitt Benckiser France that it was not possible to obtain the signature of Annick Canelas and that she may have moved.
- 7. This information prompted me to do a search in the pagesjaune.fr database for a possible new address. This database provides telephone numbers and addresses throughout France. I obtained no results for Ms. Canelas' specific locality, department, district or region. I also obtained no results in an expanded search without a specific address or first name. Attached, as Exhibit N-2, is a copy of a printout of the search that was done on 4 February 2003.
- 8. When I ran this pagesjaune.fr search, I did not appreciate the fact that clicking on the Union Jack symbol would result in an English language version. I ran a corresponding English language search on 20 February 2003, with essentially the same results. There were no hits for Annick Canelas. Attached, as Exhibit N-3, is a printout of this search.
- 9. In further e-mail correspondence with Ms. Grant and Ms. Leboucher-Madec, I learned on 7 February 2003 that Ms. Canelas had made a written statement to the effect that she would not sign anything for Reckitt & Colman.

I declare that all statements made herein of my knowledge are true and all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. Section 1001 and that such willful false statements may jeopardize the validity of this application or any patents issuing thereon.

Signed at New York, New York, this 21st day of February 2003

MH Ruen

Frederick H. Rabin



Frederick P. Fish 1855-1930

W.K. Richardson 1859-1951

45 Rockefeller Plaza Suite 2800 New York, New York

Telephone 212 765-5070

Facsimile 212 258-2291

Web Site www.fr.com

20 November 2002

Mme. Annick Canelas 53, rue de Voves F-28630 Le Coudray **FRANCE**

Re:

United States Application No. 09/381,890

PCT/GB98/00950 (WO 98/44898)

DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE

Our Ref.: 08291-619001

Dear Mme. Canelas:

In order to proceed with the above-identified United States Patent Application for Project System H, it is necessary for you to execute the enclosed Combined Declaration and Power of Attorney. Attached to the Declaration is a complete copy of the Application including the written description, claims and drawings, which was published as WO98/44898 on 15 October 1998.

I realize that, in the past, you have refused to sign the application, but we would like to remind you that you are under obligation to sign the declaration by virtue of your continuing contractual obligations with Reckitt & Colman France. You have already assigned your rights in this application to Reckitt & Colman France. The enclosed declaration is a formal requirement under the patent laws of the United States.

We are also enclosing a return envelope for your convenience. Since we are not able to obtain stamps for the return envelope, I am also enclosing ten € in cash in order to cover return postage.

I would very much appreciate your signing the enclosed Declaration without detaching WO98/44898, and returning it to us as soon as possible.

Also, we would be grateful if you would keep us informed of any changes to your address. If your address has changed from that noted on the attached declaration, please update address details on the form in manuscript.



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Mme. Annick Canelas 20 November 2002 Page 2

I thank you in anticipation of your prompt attention to this matter and look forward to receiving the executed document as a matter of extreme urgency.

Yours very truly,

Frederick H. Rabin

FHR/myw

Enclosure

cc: Ms. Amanda Wilde, Reckitt Benckiser plc, Ref. 10456P2USw

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| INTERNATIONAL APPLICATION PUBLIS | HED ! | UNDER THE PATENT COOPERATION TREATY (PCT) |
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| (51) International Patent Classification 6: | | (11) International Publication Number: WO 98/44898 |
| A61K 7/155 | A1 | (43) International Publication Date: 15 October 1998 (15.10.98) |
| (21) International Application Number: PCT/GB (22) International Filing Date: 30 March 1998 (30) Priority Data: 97400811.2 9 April 1997 (09.04.97) (34) Countries for which the regional or international application was filed: 9711447.4 4 June 1997 (04.06.97) | 30.03.9 | court (FR). DAHMS, Gerd [DE/DE]; Beerenbusch 11, D-42549 Velbert (DE). DESMOTS, Sarah [FR/FR]; 4, rue de la Fulda, F-28100 Dreux (FR). DELAGNEAU, Hubert [FR/FR]; 14, rue des Tilleuls, Bailleau l'Eveque, F-28300 Mainvilliers (FR). EP (74) Agents: ILOTT, Elizabeth, Anne et al.; Reckitt & Colman plc, |
| (71) Applicant (for all designated States except MN US): R & COLMAN FRANCE [FR/FR]; 15, rue Ampè Postale 83, F-91301 Massy Cedex (FR). (71) Applicant (for MN only): RECKITT & COLMAN PRO LIMITED [GB/GB]; One Burlington Lane, Lon 2RW (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): GUILLAUME [FR/FR]; 3, impasse de la Chesnaye, Craches, Prunay en Yvelines (FR). LEDON, Philippe [FR hauts de Chateau, F-28300 Saint Prest (FR). CA Annick [FR/FR]; 53, rue de Voves, F-28630 Le (FR). ACHER, David [FR/FR]; 1, rue Jean Lai partement 10, F-28000 Chartres (FR). HEMERY, | E, Brun F-786 VFR]; NELA Coudr illet, A | GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published With international search report. |

(54) Title: DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE

(57) Abstract

A depilatory composition in the form of an aqueous gel comprises a substance capable of degrading hair keratin and a water-soluble polymeric binder and is buffered to a pH of from 10.5 to 13.0. The polymeric binder is formed from a first component that is a charged cross-linked polymer and a second component that comprises a linear non-ionic and/or charged polymer. The composition is preferably in the form of a stable gel that can be applied by means of a pump spray.

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WO 98/44898 PCT/GB98/00950

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DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE

This invention relates to depilatory compositions, in particular those in the form of a gel.

5 Compositions for removing superfluous body hair are well known. These are of various types. type of composition is in the form of a cream containing a depilatory agent which degrades hair keratin. In the case of a depilatory cream, hair is 10 removed by applying the cream to an area of skin to be depilated, leaving the cream on the skin for sufficient time for the hair to be chemically dissolved, and then wiping and/or washing the cream and unwanted hair from the skin. Another type of composition is in the form of a wax, which is 15 initially heated before being applied to the skin in a generally molten state. It is then allowed to solidify before being removed from the skin together with unwanted hair.

Dritish patent specification no. GB 2 223 170

discloses a two-part depilatory composition in which
the first part comprises polyvinyl alcohol (PVA) and
the second part comprises borax and a polyhydroxy
alcohol, or Congo red; the second part functioning to
effect a controlled rate of cross-linking of the PVA.

After in situ cross-linking, such compositions
solidify, requiring the use of a plasticiser to
enable the solid to be peeled from the skin. Such
compositions preferably also include a surfactant.

Depilatory compositions in the form of a gel are less common and attempts to market such gels have had

only limited success. The gels tended to have a higher skin irritancy than cream depilatories and became very sticky when being rinsed off because of the nature of the polymer present in the formulation. Moreover, depilatory compositions need to be of high pH, and problems were encountered in formulating a gel which would be stable in such highly alkaline conditions.

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It is an aim of the present invention to provide a depilatory composition in the form of a gel which does not suffer from these disadvantages.

It is a further aim of the present invention to provide a depilatory composition in the form of a gel which may be sprayed onto the skin without propellant.

It is another aim of the present invention to provide a depilatory gel having a reduced tendency to irritate the skin.

It has been found that these aims can be achieved by forming a gel from two water-soluble polymeric components and in a pH environment maintained by a preferred buffer system. One of the polymeric components provides the gel with appropriate viscosity and the other polymeric component contributes to the stability of the gel.

Accordingly, one aspect of the present invention provides a depilatory composition in the form of an aqueous alkaline gel comprising:-

- (i) a first polymeric binder comprising a charged, cross-linked, water-soluble polymer;
- (ii) a second polymeric binder comprising a linear, water-soluble polymer;
 - (iii) a depilatory agent; and
- (iv) a buffer system capable of maintaining the pH of the composition at a value of from 10.5 to 13.0.

Preferably, the composition remains in the form of an aqueous alkaline gel in use.

- According to another aspect of the present invention, there is provided a process for producing a depilatory composition, which process comprises:-
- (i) forming a first premix by mixing water and a first polymeric binder comprising a charged, cross-linked, water-soluble polymer; forming a second premix by mixing water, a buffer and a second polymeric binder comprising a linear, water-soluble polymer; and optionally forming a third premix by mixing a depilatory agent and a keratin degradation accelerator;
 - (ii) adding a depilatory agent or the third premix to the second premix with stirring;

(iii) adding the first premix to the resultant mixture with stirring; and

(iv) adjusting the pH to between 10.5 and 13.0.

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The depilatory compositions of the present invention accordingly possess unique rheological qualities, which can be defined inter alia by their characteristic viscosity properties. For example, the shear rate (in seconds⁻¹) needed to decrease the viscosity of the gel by half is in the range of from about $10^{-2} \, \mathrm{s}^{-1}$ to about 200 s^{-1} , preferably from about 1 s⁻¹ to about 50 s⁻¹. Such a large drop in the viscosity of depilatory compositions is not seen with prior art formulations over these shear rate ranges. At a low shear rate of about 10⁻² s⁻¹, the viscosity of the compositions of the present invention is of the order of 103 Pas (106 cP), whereas at a high shear rate of about 500 s⁻¹, their viscosity is of the order of about 0.1 Pas (100 cP). At a very high shear rate of the order of 4×10^4 s⁻¹, such as that effected by a spray-pump (see Table 2 below), it is extremely difficult to measure viscosity, but it can be envisaged that that of the compositions of the present invention would be very low.

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Preferably, a gel in accordance with the present invention has unique viscosity and elasticity, sustained high ionic strength and high pH.

The cross-linked polymer of the first component is such that it swells at high pH (pH 10.5 to 13) and preferably has only carbon-carbon bond

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cross-linkages. More preferably, the elastic modulus of the first component is in the range of from 200 Pa to 1000 Pa, conveniently, 400 Pa to 600 Pa, when measured at a frequency of 10 rad/s; at a temperature of 20°C and a strain of 0.1. The elastic (storage) modulus (G') corresponds to the energy which can be stored and released by the material.

Suitable first components include a cross-linked acrylic polymer such as a carbomer, for example, Ultrez 10 and/or Carbopol ETD 2020 from Goodrich, and Structure 2000 and 3001 from National Starch.

Another suitable polymer is a cross-linked polyacrylamide such as Seppigel 305. Most preferably, the polymer of the first component is a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene available from ISP under the trade designation Antaron Stabilise 06.

Preferably, the depilatory composition includes from 0.1% to 3.0% by weight of the first component.

The second component is a linear non-ionic polymer and/or a positively charged polymer and/or a negatively charged polymer. The second, linear component is one which acts as a suspending agent for the gel and moderates the elastic modulus thereof to enable spreadability. Any suitable linear polymer may be chosen which enables the gel structure to be maintained at the osmotic pressure in the composition. Such linear polymers include hydrophilic polymers having a molecular weight of less than about 600 and non-ionic gel-forming polymers. For example, the second component may

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comprise a water-soluble non-ionic polymer such as polyethylenimine, polyethylene oxide or polyethylene glycol (PEG), a hydrophobically-modified polyol such as Acrysol 44 from Rohm and Haas, or a polyvinyl pyrrolidone such as PVP K from ISP or Luviskol from BASF.

Alternatively, or additionally, the second component may comprise a natural water-soluble polymer such as cellulose, hydroxypropyl cellulose, cetyl hydroxyethyl or propyl cellulose (for example Natrosol Plus from Aqualon), starch, polyglucoside, a polysaccharide such as an alginate, guar gum, carrageenan (lambda, kappa, iota), a sclerotium gum, a polypeptide with or without any alkyl chain (C1 to C18) modification and cationic end groups such as quaternary ammonium groups with counter ions such as chlorides and bromides, or a copolymer of sodium acrylate and dimethyl diallyl ammonium chloride (such as that known as Merquat), or a copolymer of hydroxy ethyl cellulose and dimethyl diallyl ammonium chloride (such as that known as Celquat).

Most preferably, the second component comprises polyethylenimine (such as that known under the trade name Lupasol from BASF) and/or polyvinyl pyrrolidone (such as that known under the trade designation PVP K-30 from ISP).

Preferably, the depilatory composition includes from 0.2% to 3.0% by weight of the second component.

The first and second components, when combined in accordance with the present invention, form a

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polymeric binder having unique viscosity and elasticity.

The depilatory agent may comprise any substance capable of degrading keratin and may be, for example, a sulphur compound such as potassium thioglycolate, dipotassium thioglycolate or thioglycerol.

Preferably, the depilatory composition includes from 0.1% to 6.0% by weight of depilatory agent, preferably from about 3.0% to 5.0%.

Also, the composition may optionally include component(s) which will accelerate the keratin reduction reaction such as urea, thiourea or dithioerythritol, dimethyl isosorbide (DMI), ethoxydiglycol (Transcutol) and methyl propyldiol (MP diol). DMI is preferred.

It is important that the pH of the composition is in the range of from about 10.5 to 13, and preferably about 11.0 to 12.5, to provide good depilation within about 5 minutes. In order to achieve this, a suitable buffer should be present, such as sodium silicate and/or L-arginine, sodium nicotinate and potassium polyethylenimine or a phosphate.

Generally, the buffer system will be present in an amount of from about 2.0% to 8.0% by weight, preferably from about 2.0% to 4.0% by weight.

Advantageously, the buffer system comprises from 2.0% to 3.0% by weight of L-arginine and from 0 to 1.0% by weight of polyethylenimine.

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The pH buffer system preferably ensures efficacy of the depilation within 5 minutes whilst also minimizing skin irritation.

- In order to achieve minimum skin irritation, a composition including L-arginine and/or polyethylenimine, urea and potassium and/or dipotassium thioglycolate is preferred.
- In accordance with a preferred depilatory composition of the present invention, there is provided an aqueous alkaline gel comprising:
 - (i) 0.1% to 3.0% w/w of a first polymeric binder comprising a charged, cross-linked, water-soluble polymer, preferably a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene;
- (ii) 0.2% to 3.0% w/w of a second polymeric
 binder comprising a linear, water-soluble
 polymer, preferably polyethylenimine
 and/or polyvinyl pyrrolidone;
- - (iv) 2.0% to 6.0% w/w, preferably 2.0% to
 4.0% w/w of a buffer system, preferably
 L-arginine and/or polyethylenimine; and,
 optionally,

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(v) 5.0% to 10.0% w/w of an acelerator for the degradation of keratin, preferably urea;

whereby the composition preferably remains in the form of an aqueous alkaline gel in use.

The gels according to the present invention are preferably transparent and remain transparent in use, depending on the amount of active ingredients present and on the procedure of mixing. Generally, they have a thick, rich feel and excellent rinsability with water. Moreover it is not necessary to include a surfactant in the compositions of the present invention, and hence they are less likely to cause skin damage.

15 Due to the special rheology of the polymeric binder, it is possible to include beads, dyes and/or oily particles which will remain dispersed in the gel. Optional excipients include compounds for soothing the skin such as azulene or jojoba and polyethylene glycol ester to reduce irritation. 20 Suitable beads include encapsulated algae extract and sweet almond oil (Elespher). It is also possible to include hydrating, accelerating and/or reducing agents in beads. For example, water-soluble materials such as urea, potassium thioglycolate or 25 thioglycerol can be included in beads, so that these actives are encapsulated.

Although it is possible to provide a gel in accordance with the present invention that has visco-elastic properties suitable for spraying, the depilatory material may alternatively be applied by

means of a roll-on device or in conventional shower gel packaging.

Other ingredients may be included in the compositions of the invention, such as are known in the art.

Unless otherwise stated, all percentage weights referred to herein are % weights based on the weight of the total composition.

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For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:-

- Figure 1 is a graph of shear stress (Pa) against shear rate (s⁻¹) for a series of polymeric binders (first component) for the composition of the invention;
- Figure 2 is a graph of the yield points (Pa) of the binders of Figure 1 against the cross-linked polymer (first component) contents (% w/w) of the binders;

Figure 3 is a graph of viscosities (Pas) of the binders of Figure 1 against the shear rate (s⁻¹) of the binders; and

Figure 4 is a graph of the elastic (G') and viscous (G'') moduli against stress/strain (Pa) of the first component of the composition.

The following Examples illustrate the invention:

EXAMPLES 1 TO 6

A series of depilatory gels was prepared having the compositions shown in the following Table 1:

TABLE 1

| | TABLE 1 | | | | | | |
|----|-----------------|-------|-------|--------|-------|-------|-------|
| | | 1 | 2 | 3 | 4 | 5 | 6 |
| | Phase 1 | | | | | | |
| | Polymer A | 3.0% | 2.5% | 2.25% | 2.0% | 1.5% | 1.0% |
| 10 | deionised water | 47.0% | 39.0% | 36.35% | 31.4% | 23.5% | 15.7% |
| | Phase 2 | | | - | | | |
| | Polymer B | 2.0% | 2.0% | 2.0% | 2.0% | 2.0% | 2.0% |
| | pH buffer | 3.0% | 3.0% | 3.0% | 3.0% | 3.0% | 3.0% |
| 15 | deionised | 10.7% | 19.2% | 24.8% | 26.4% | 38.3% | 45.8% |
| 13 | water | | | | | , | |
| | Phase 3 | | | · | | | |
| | potassium | 14.7% | 14.7% | 14.7% | 14.7% | 14.7% | 14.7% |
| | thioglycolate | | | | | | |
| 20 | (30%) | | | | | | |
| 20 | urea | 8.0% | 8.0% | 8.0% | 8.0% | 8.0% | 8.0% |
| | Final mix | | | | | | |
| | Phase 1 | 50.0% | 41.5% | 37.5% | 33.4% | 25.0% | 16.7% |
| | Phase 2 | 15.7% | 24.2% | 29.8% | 33.4% | 43.3% | 50.8% |
| 25 | Phase 3 | 22.7% | 22.7% | 22.7% | 22.7% | 22.7% | 22.7% |
| | Potassium | 11.6% | 11.6% | 10.0% | 10.5% | 9.0% | 9.8% |
| | hydroxide 50% | | | | | | |
| | Total | 100% | 100% | 100% | 100% | 100% | 100% |
| | Total deionised | 57.5% | 58.0% | 59.9% | 59.6% | 1.6% | 61.3% |
| 30 | water | | | | | | |

All amounts are expressed as % weight/weight total composition. Polymer A was a methyl vinyl ether/maleic anhydride copolymer cross-linked with dodecadiene known under the trade designation Antaron STO6 from ISP; and polymer B was polyvinyl pyrrolidone known under the trade designation PVP K-30 from ISP. The pH buffer system was a combination of 2% w/w of L-arginine and 1% w/w of polyethylenimine.

In each case, an Olsa mixer with planar stirring and a high shear stirrer equipped with a vacuum and heating/cooling system was used. The procedure was as follows:

Phase 1: Premix polymer A and water.

The water was placed in the mixer and polymer A
was added slowly with stirring. When the polymer had
been dispersed, heating was commenced whilst
maintaining a vacuum of about 0.5 to 0.8 bar to avoid
air bubbles. The vacuum pump was operating
intermittently in order to reduce water loss. When
the temperature had reached about 80°C an almost
clear gel had been formed. This was allowed to cool
and swell overnight.

Phase 2: Premix polymer B, water, pH buffer

The water and polymer B were introduced into the mixer and homogenised for 2 minutes. The pH buffer system was then added and the mixture homogenised for a further 2 minutes.

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Phase 3: Urea and potassium thioglycolate

The urea was added to the potassium thioglycolate in a beaker and stirred for 1 hour to complete dissolution.

-13-

Final mixing: The final mixing was effected as
follows:-

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Phase 2 was placed in the vessel and phase 3 was added with stirring for 15 minutes. A white precipitate appeared. Phase 1 was added while mixing slowly under vacuum (0.5 bar) with planar stirring and the high shear stirrer operated at low speed. The mixture was homogenised for 30 minutes until a lumpy gel had been formed. The potassium hydroxide was then added. The gel became clear and transparent. The pH was adjusted, as necessary, to from 11.0 to 12.5.

Each of the resultant gels was subjected to rheological measurement. The parameters considered were shear stress, shear rate, viscosity and yield point. The results are shown in the Figures.

As can be seen from Figure 1, the variation of shear stress with shear rate is non-linear between 0 and 500 s⁻¹ and thus the gel has a non-Newtonian behaviour with a yield point. The yield point is the stress required to make the gel flow. Figure 2 shows that the variation of the yield point (TauO), i.e. the shear stress for zero shear rate, varies exponentially. For a small variation in the amount of polymer A, the variation in the yield point, i.e. the variation in spreadability or consistency, may be significant.

The following table, Table 2, gives characteristic shear rates of products to be sprayed with a conventional pump spray and products to be applied from a shower gel packaging.

5 TABLE 2

| Type of packaging | Flow Rate (from packaging) of product (cm ³ /s) | Radius of the external output (cm) | Shear rate (s ⁻¹) |
|----------------------|--|--|----------------------------------|
| 0 Pump Spray | 0.17 | 0.0175 (radius of the actuator) | 500-40000 |
| Shower gel packaging | 8.8 | ca 0.15 | 500-30000 |

The relationship between shear rate and viscosity is shown in Figure 3, from which it can be seen that, at the shear rates given in Table 2, the viscosity is very low.

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It has been found that, in order for the gel to be sprayable with a pump spray, its yield point should be not more than from 100 to 200 Pa. If, however, the gel is to be used in a shower gel pack, the yield point may be as high as 300 Pa. Also, in order for the product to remain still for 5 minutes on the skin when held vertical, the yield point needs to be about 60 Pa or more. Thus gels wherein polymer A constitutes from 2.0% to 2.5% w/w are preferred for spraying, whereas rather lower percentages of polymer A may be present where the gel is to be used in the form of a shower gel packaging.

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In a further series of experiments, the blue powder Azulene was dissolved in thick paraffin oil to produce a 1% w/w solution. 2% w/w of this solution was then slowly added to each of the above gels and stirred for 5 minutes. The Azulene remained stably suspended within the gel in the form of blue oily droplets. Depending upon the concentration of Azulene in the premix and the concentration of premix in the gel, the size and colour of the droplets and the overall transparency of the gel could be adjusted.

In a further series of experiments, 0.5% w/w of Elespher was slowly added to each gel and stirred for 5 minutes. Elespher consists of encapsulated beads of algae extract and sweet almond oil, and is green in colour. The beads were stably suspended in the gel.

EXAMPLE 7

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Gels were made in accordance with the following Table 3:

TABLE 3

| 25 | <u> </u> | 8w/w |
|----|---------------|------|
| | Phase 1 | |
| | polymer A | 1-3 |
| 30 | water | 40.0 |
| | KOH 50% | 4.0 |
| | Total phase 1 | 47.0 |

| | Phase 2 | | | |
|----|---------------------|----------|--|--|
| | polymer B | 0.6-3 | | |
| | buffer system | 3.0 | | |
| 5 | water | 8.2 | | |
| | Total phase 2 | 11.8 | | |
| | Phase 3 | | | |
| | water | 6.5 | | |
| 10 | potassium | 14.7 | | |
| | thioglycolate (30%) | | | |
| | Total phase 3 | 29.2 | | |
| | water | to 100.0 | | |
| | кон 50% | 6.15 | | |

The amount of water was varied depending upon the amounts of polymers A and B present. Polymers A and B were as used in Examples 1 to 6 and the mixing technique employed was as described in those Examples. In each case, stable, sprayable depilatory gels were obtained. This showed that for a range of polymer A between 1.0% and 3.0%, a range of polymer B between 0.6% and 3.0% could be used and a stable, sprayable gel formed.

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CLAIMS

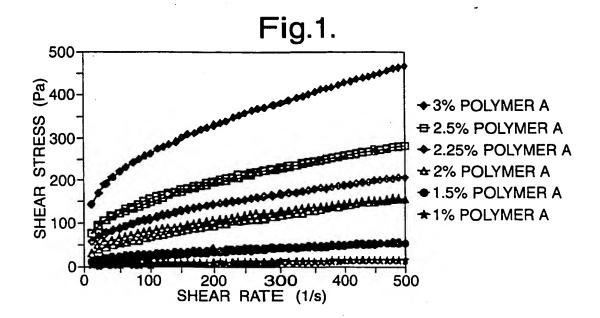
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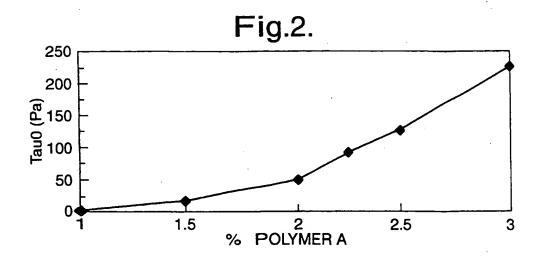
- A depilatory composition in the form of an aqueous alkaline gel comprising:
- 5 (i) a first polymeric binder comprising a charged, cross-linked, water-soluble polymer;
- (ii) a second polymeric binder comprising a
 linear, water-soluble polymer;
 - (iii) a depilatory agent; and
- (iv) a buffer system capable of maintaining the pH of the composition at a value of from 10.5 to 13.0.
 - 2. A depilatory composition as claimed in claim 1, wherein the cross-linked polymer of the first component is a cross-linked acrylic polymer, a cross-linked polyacrylamide or a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene.
- A depilatory composition as claimed in claim 1 or
 which includes from 0.1 to 3.0% w/w of the first
 component.
 - 4. A depilatory composition as claimed in any of claims 1 to 3, wherein the second component comprises a linear, non-ionic polymer and/or positively charged polymer and/or negatively charged polymer.

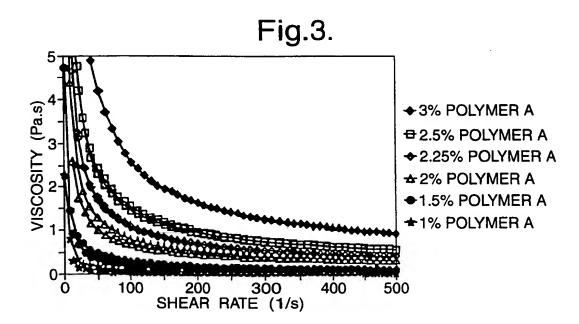
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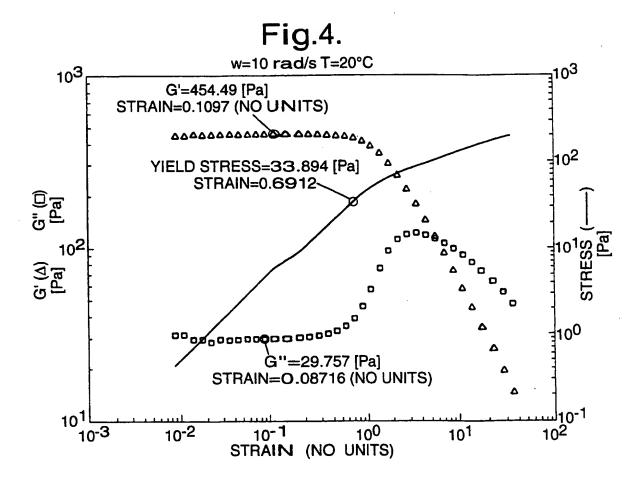
- 5. A depilatory composition as claimed in claim 4, wherein the second component comprises one or more of polyethylenimine, polyvinyl pyrrolidone, polyethylene oxide, polyethylene glycol, cellulose, hydroxy propyl cellulose, cetyl hydroxy ethyl cellulose, cetyl hydroxy propyl cellulose, starch, a polyglucoside, a polysaccharide, guar gum, carrageenan, a sclerotium gum, a polypeptide, and a copolymer of sodium acrylate or of hydroxy ethyl cellulose with dimethyl diallyl ammonium chloride.
- 6. A depilatory composition as claimed in any preceding claim, which includes from 0.2 to 3.0% w/w of the second component.
- 7. A depilatory composition as claimed in any preceding claim, wherein the depilatory agent is potassium thioglycolate or thioglycerol.
- 8. A depilatory composition as claimed in any preceding claim, including an accelerator for the degradation of keratin, such as urea, thiourea, dithioerythritol, ethoxydiglycol or methylpropyldiol.
 - 9. A depilatory composition as claimed in any preceding claim, including a buffer system comprising a silicate, arginine such as L-arginine, a nicotinate, polyethylenimine or a phosphate.
 - 10. A depilatory composition as claimed in any preceding claim and having a yield point of not more than 200 Pa.

- 11. A depilatory composition as claimed in any preceding claim, wherein the shear rate needed to decrease the viscosity of the gel by half is in the range of from 10^{-2} s⁻¹ to 200 s⁻¹, preferably from 1 s⁻¹ to 50 s⁻¹.
- 12. A depilatory composition as claimed in any preceding claim, in the form of a sprayable gel.
- 13. A process for producing a depilatory composition as claimed in any preceding claim, which process comprises:-
 - (i) forming a first premix by mixing water and the first component; forming a second premix by mixing water, a buffer and the second component; and optionally forming a third premix by mixing a depilatory agent and a keratin degradation accelerator;
- (ii) adding a depilatory agent or the third premix to the second premix with stirring;
 - (iii) adding the first premix to the resultant
 mixture with stirring; and
 - (iv) adjusting the pH to between 10.5 to 13.0.









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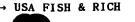
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Attorney's Docket No.: 08291-619001 Client's Ref. No.: 10456P2-Usw

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I Annick CANELAS hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor, together with Bruno GUILLAUME, Philippe LEDON, David ACHER, Séverine POETTE, Gerd DAHMS, Sarah DESMOTS and Hubert DELAGNEAU, of the subject matter which is claimed and for which a patent is sought on the invention entitled DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE, the specification of which:

was filed on <u>September 24, 1999</u> as Application Serial No. <u>09/381,890</u> and, was described and claimed in PCT International Application No. <u>PCT/GB98/00950</u> filed on <u>30 March 1998</u>.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information I know to be material to patentability in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

| Country | Application No. | Filing Date | Priority Claimed |
|----------|-----------------|--------------|------------------|
| European | 97400811.2 | 9 April 1997 | [X] Yes [] No |
| British | 9711447.4 | 4 June 1997 | [X] Yes [] No |

I hereby appoint the following attorneys and/or agents to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or

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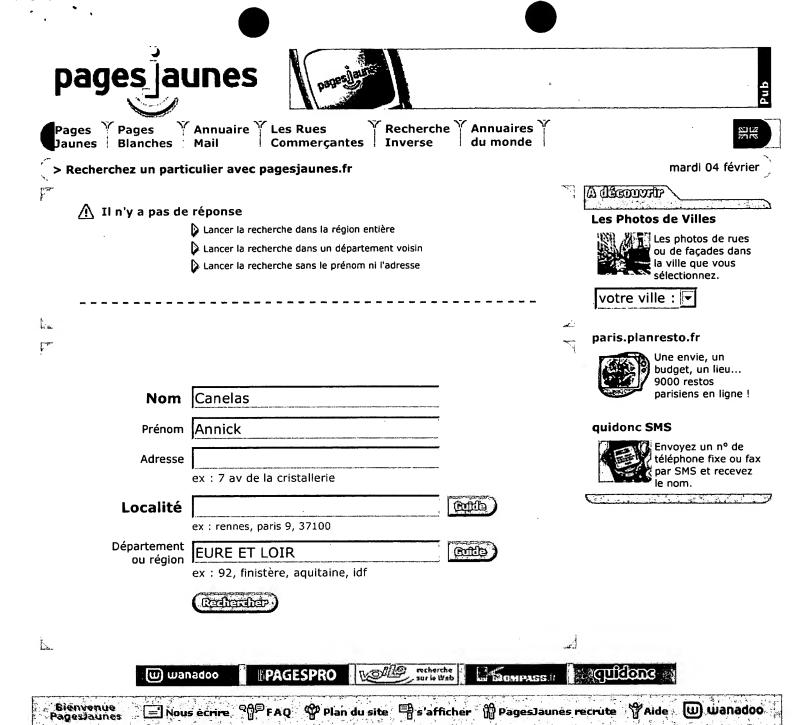


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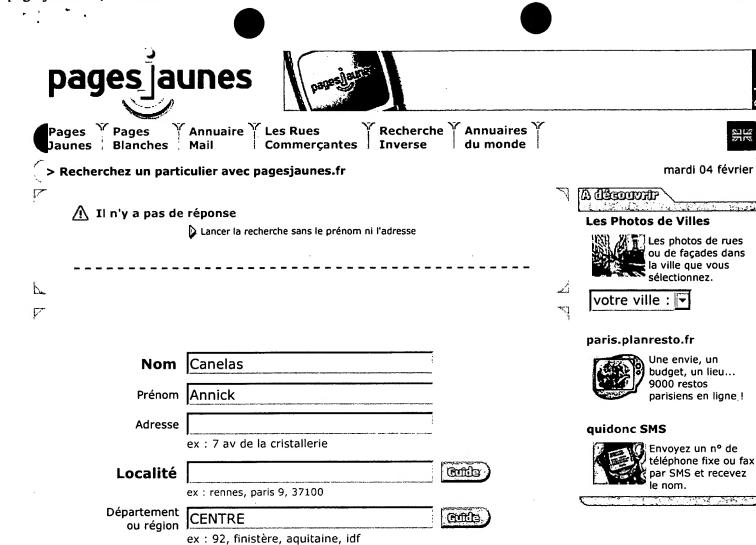


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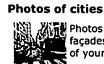
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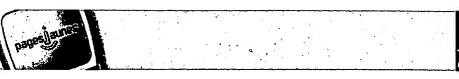
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- COUDRAY (53200)
- COUDRAY (45330)
- COUDRAY (27150)
- LE COUDRAY MONTCEAUX (91830)
- LE COUDRAY SAINT GERMER (60850)
- LE COUDRAY MACOUARD (49260)
- COUDRAY RABUT (14130)
- COUDRAY AU PERCHE (28330)
- COUDRAY BAILLET (56)
- LE COUDRAY SUR THELLE (60790)
- BOIS DU COUDRAY (95)
- LE COUDRET (17)
- LA COUDRE (79150)
- COUDREE (74)
- COUDRES (27220)
- SAINT MARTIN DE LA COUDRE (17)
- GISAY LA COUDRE (27330)
- SAINT GERMAIN DE LA COUDRE (61130)
- SAINT AUBIN DES COUDRAIS (72400)
- SAINT DENIS DES COUDRAIS (72110)

1

Name | Canelas

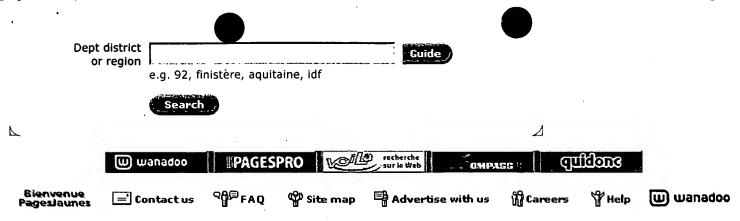
First name | Annick

Address

e.g. 7 av de la cristallerie

Town

LE COUDRAY e.g. rennes, paris 9, 37100 Off b

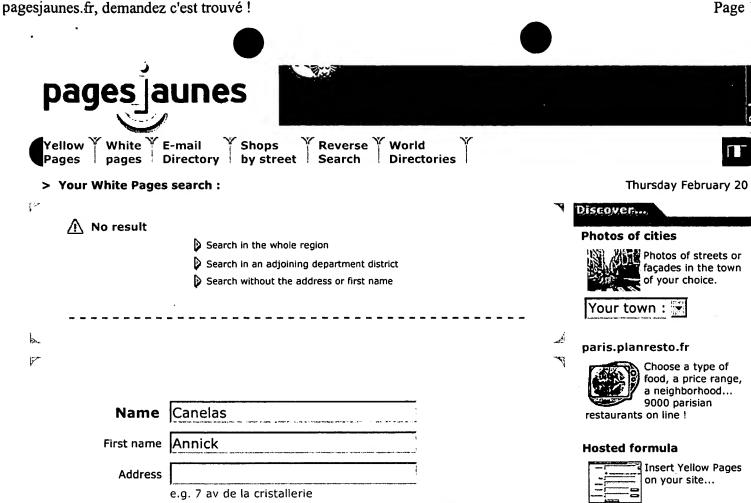




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| First name Annick | |
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> Your White Pages search:

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CANELAS Your query:

EURE ET LOIR (28)

> DREUX (28100)

Canelas Fernando

Recherche multi-départements

7 answer(s) with exact spelling

(list 1-7)

02 37 42 75 79

02 37 50 02 75

Annuaire

Useful



Les Pages Jaunes d'Indre et Loire

Query:

&Pages Jaunes

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Activity:

Canelas Horacio

11 r St Martin 28100 DREUX | Map

27 av Prés John Kennedy 28100 DREUX | Map

LOIR ET CHER (41)

> LA FERTE BEAUHARNAIS (41210)

Canelas Acacio

429 r Gén Alexandre 41210 LA FERTE BEAUHARNAIS 02 54 83 72 98

Canelas Antonio

66 r Avrain de Meung 41210 LA FERTE BEAUHARNAIS

02 54 83 66 40

Canelas Georges

253 r Gén Alexandre 41210 LA FERTE BEAUHARNAIS

02 54 83 74 85

> BLOIS (41000)

Canelas-Reis Maria

20 r Christophe Colomb 41000 BLOIS | Map

02 54 43 87 69

LOIRET (45)

> PITHIVIERS (45300)

Canelas Arlindo

1 sq Marsainvilliers 45300 PITHIVIERS

02 38 30 35 73

> Answers with approximate spelling

14 answer(s)

(list 1-10)

CHER (18)

> CHATEAUMEILLANT (18370)

Canellas Florian



3 r Rouettes 18370 CHATEAUMEILLANT

02 48 61 47 82

> GENOUILLY (18310)

Canellas Jacques

Les Poëlets 18310 GENOUILLY

02 48 52 22 41

> SAINT GEORGES SUR LA PREE (18100)

Canellas Gérard

9 r La Prée 18100 SAINT GEORGES SUR LA PREE

02 48 52 02 33

EURE ET LOIR (28)

> LUCE (28110)

Canéla Daniel

11 r Bruxelles 28110 LUCE | Map

02 37 35 37 53

> MAINVILLIERS (28300)

Canela Sabine

pl Jean Macé 28300 MAINVILLIERS | Map

02 37 20 09 85

INDRE (36)

> CHABRIS (36210)

Canelhas Antonio

4 r Chauchy 36210 CHABRIS

02 54 40 18 72

Canelhas Carlos

79 r Varennes 36210 CHABRIS

02 54 40 03 45

LOIR ET CHER (41)

> GIEVRES (41130)

Canelhas Georges

7 r Hubardière 41130 GIEVRES

02 54 98 61 43

> PONTLEVOY (41400)

Canella Omer

18 r Colonel Filloux 41400 PONTLEVOY

02 54 32 51 83

fax : **02 54 32 61 28**

> SELLES SUR CHER (41130)

Canelhas Albino

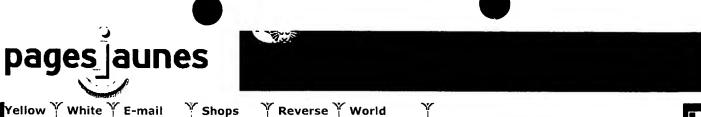
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02 54 97 65 45

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New search:

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| | | Name | Canelas | | | | |
| | | First name | | | **** | | |
| | | Address | | | | | |
| | | Town | | | the said the relative as day. | | |
| | | Dept district or region | CENTRE | | | | |
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CANELAS Your query:

Recherche multi-départements

> Answers with approximate spelling

14 answer(s)

(list 11-14)

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Query:

LOIRET (45)

> CHAILLY EN GATINAIS (45260)

Canellas Frédéric

10 chem Bourcheveaux 45260 CHAILLY EN GATINAIS

02 38 96 22 50

> CORBEILLES (45490)

Canela Maxime

4 r Bourg Neuf 45490 CORBEILLES

02 38 92 22 02

fax: 02 38 96 48 93

Canela Maxime

30 r Bréau 45490 CORBEILLES

02 38 92 93 04

> FLEURY LES AUBRAIS (45400)

Canelhas Luis

13 av Cosmonautes 45400 FLEURY LES AUBRAIS | Map

02 38 70 04 16



New search:

| Name | Canelas |
|-------------------------------------|---------|
| First name | |
| Address | |
| | |
| | |
| Town | |
| | |
| Town Dept district or region | CENTRE |

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Attorney's Docket No.: 08291-619001 / 10456P2-Usw

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:

Sarah DESMOTS et al.

Int'l Appln. No.

PCT/GB98/00950

Int'l Filing Date:

30 March 1998

U.S. Serial No.:

09/381,890

Title:

DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE

DECLARATION RE INVENTOR ANNICK CANELAS

I, Karen R. Grant, declare as follows:

- 1. I am a Formalities Officer in the Group Patents Department of Reckitt Benckiser plc.
- 2. Previous attempts to obtain a Combined Declaration and Power of Attorney from Annick Canelas were unsuccessful. These attempts were documented as Exhibits C and F attached to the Declaration of Amanda J. Wilde of 16 March 2001, and further documented as Exhibit I attached to Ms. Wilde's Declaration of 18 February 2002 which was filed herein on 21 February 2002.
- 3. On 20 November 2002, Mr. Frederick H. Rabin made another attempt to secure Ms. Canelas' signature. Specifically, he sent her a new Combined Declaration and Power of Attorney, which was attached to a copy of PCT published application WO 98/44898. A copy of Mr. Rabin's letter to Ms. Canelas was sent to Ms. Amanda Wilde, my predecessor, and was received by me.
- 4. On 17 December 2002, Mr. Rabin informed me that the material sent Ms. Canelas was not returned as undeliverable and that Ms. Canelas did not respond in any way to said letter. Mr. Rabin asked my assistance in securing the signature of Ms. Canelas.
- 5. Subsequently, I had a number of telephone and e-mail exchanges with Ms. Christelle Leboucher-Madec, a legal assistant in Reckitt Benckiser France, our affiliated company. Ms. Leboucher-Madec informed me that it was not possible to contact Ms. Canelas. However, on 7 February 2003, Ms.

Attorney's Docket No.: 08291-619001 / 10456P2-Usw

Leboucher-Madec informed me that, in February of 2002, she had received a note from Ms. Canelas to the effect that she would not sign any documents on behalf of Reckitt & Colman. A copy of this note is attached as Exhibit N-4.

6. Ms. Canelas' note was in French and I had it translated. A copy of the translation is attached hereto as Exhibit N-5.

I declare that all statements made herein of my knowledge are true and all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. Section 1001 and that such willful false statements may jeopardize the validity of this application or any patents issuing thereon.

Signed at Kingston upon Hull, England, this 21st day of February 2003

allm

Karen R. Grant